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IN REPLY TO RFP CC NO

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November 1, 1994 94-RF-11105

Norma I Castaneda
Environmental Restoration Division
DOE/RFFO

TRANSMITTAL OF PHASE I RESULTS FOR THE CHEMICALLY ENHANCED STEAM STRIPPING TREATABILITY STUDY - WSB-122-94

Action None

The purpose of this letter is to transmit the draft Phase I Report for the Chemically Enhanced Steam Stripping Treatability Study (Attachment 1) to the Department of Energy (DOE) EG&G Rocky Flats, Inc transmitted the draft report by fax to yourself and Mike Harris on October 31, 1994 Please submit only major comments since an edited draft copy of this report will be submitted to DOE for review on November 7, 1994

Any questions or comments should be directed to Virginia Ferguson of the OU 2 Closure Group, Treatability Studies/Feasibility Studies Team at extension 8752

WSBusby

W S Busby
Manager, OU-2 Closure
Environmental Restoration Program Division

VLF pap

Attachment
As Stated

cc
M J Harris - NFT, Inc

ADMIN RECORD

BZ-A-00128

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PHASE I REPORT

Chemically Enhanced Steam Stripping of Radionuclides from Rocky Flats Plant Soils

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1.0 ABSTRACT/EXECUTIVE SUMMARY

A series of experiments were performed at Los Alamos National Laboratory to identify leaching schemes that would be suitable for leaching plutonium and americium from the contaminated soil. The procedure for these batch desorption experiments on RFP soil (contaminated with plutonium and americium) is described and preliminary analysis of the results is presented. This project combines the concepts of redox, chelation, and stream processing to treat plutonium and americium contaminated soils which were collected from 903 Pad Area of Rocky Flats Plant. The goal is to conduct bench scale experiments of this soil extraction process for removal of radionuclides from actinide contaminated soils. The objective of this work is to provide treatability data to be used in screening and selecting remedial processes for treatment of Pu and Am contaminated soils in RFP. This report is the Phase I progress report for the treatability study project "Chemically Enhanced Steam Stripping of Radionuclides from Rocky Flats Plant (RFP) soils".

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2.0 INTRODUCTION

2.1 Site History

The particular focus in this study is Operational Unit 2, 903 Pad area which potentially contains plutonium, americium, and organic solvents. Experiments were performed on soils which were collected from the 903 Pad by Rocky Flats personnel. The 903 Pad Area, encompassing the original 903 Drum Storage Site, was used from October 1958 to January 1967 for storage of radioactively contaminated oil drums whose contents were described by Calkins (1970).

"Most of the drums transferred to the field were nominal 55-gallon drums, but a significant number were 30-gallon drums. Not all were completely full. Approximately three-fourths of the drums were plutonium-contaminated, while most of the balance contained uranium. Of those containing plutonium, most were lathe coolant consisting of a straight-chain hydrocarbon mineral oil and carbon tetrachloride in varying proportions. Other liquids were involved, however, including hydraulic oils, vacuum pump oil, trichloroethylene, perchloroethylene, silicone oils. In 1959 or possibly earlier ethanolamine was added to the oil to reduce the corrosion rate of the steel drums."

An estimated 5,000 gallons of liquid (Freiberg, 1970) containing 86 g (5.3 Ci) of plutonium leaked into the soil. Site grading in preparation for applying an asphalt cap over the area included moving "slightly" contaminated soil. A total of 33 drums of radioactively contaminated rocks were removed, and two courses of clean fill material were placed over the site. The asphalt covering was applied some two months later (Freiberg, 1970). The cover is approximately 8 centimeters (cm) thick and underlain by approximately 15 cm of loose gravel and 8 cm of fill dirt.

2.2 Technology Introduction

This work evaluate the use of thermally enhanced aqueous extraction processing of soils for the removal of radionuclides. The concept combines the technologies of redox, chelation, and steam processing to meet the challenge of 903 Pad area soils. This project conducted proof-of-principle, bench-scale evaluations of chemically enhanced steam stripping for the mobilization and removal of radionuclide from contaminated soils. this effort is part of the treatability studies for remedial screening and selection process for actinide contaminated soils in OU2's 903 Pad area, but has potential applicability to other radionuclide contaminated sites at Rocky Flats Plant.

Actinides (such as Pu, Am, and U) tend to have a strong affinity for the minerals in most soils (Thomas, 1987 and Triay et al., 1991). The mechanisms that normally dominate radionuclide sorption are surface complexation (Combes et al., 1992) and ion exchange (Triay and Rundberg, 1989 and 1987). Since the radioactive metals are Lewis acids (i.e., acquire electrons to reach an inert state), complexants that act as Lewis bases (i.e., have electron pairs that can be shared with the metal) can be utilized to leach Pu and Am from contaminated soils.

The effectiveness of complexants to remove plutonium and americium from soils depends on the chemical form of these metals in the contaminated area. Pimpl and Schucttelkopf (1991) report that Pu, Am, and Cm in soil columns, contaminated with 5 mCi of each actinide near the surface, were mobilized and migrated with an irrigation solution containing 0.1M DTPA. After elution of the irrigation solution less than 3 pCi/g of activity was found in the soil. Lee and Marsh (1992) have reported that a significant amount of uranium can be extracted from Fernald soils utilizing citric acid and carbonate.

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3.0 TREATABILITY STUDY APPROACH

3.1 Test Objectives and Rationale

Testing will be conducted in three tasks: (1) select promising chelator-redox systems using fast turnaround, lab-scale, chemically enhanced steam extraction test, (2) further refining the list of chemical enhancers to the more promising chelator-redox agent systems using bench-scale, soil column washing test, and (3) optimize the most promising chelator-redox systems using bench-scale, soil column washing tests. The objectives are:

(1) To select appropriate chelating-redox agent systems, and to define test matrix and work plan for bench scale tests.

(2) To perform bench-scale tests to evaluate plutonium and americium mobilization and removal efficiency using suitable chelating agents and redox conditions.

(3) To perform bench-scale tests to optimize plutonium and americium removal using suitable conditions that minimize chemical loading and modification of soils.

This report will focus on the results obtained from bench-scale tests to evaluate plutonium and americium mobilization and removal efficiency using suitable chelating agents and redox conditions.

3.2 Treatment Technology Description

3.2.1 Treatment technology

Soil decontamination and washing evaluations have been conducted at RFP since the early 1970s and results have appeared in reports in the internal and external literature. R. L. Olsen et al. (1980) described the decontamination of the 903 Pad area following cleanup of the leaking drums in 1968. They reported that radiological contamination of 2000-300,000 dpm/100 cm^2 had penetrated the 903 Pad soils to a depth of 20 cm.

Hicks and Blakeslee (1981) summarized a decade of soil characterization and bench-scale attrition scrubbing studies performed at RFP on RFP 903 Pad Area soils. Plutonium in these soils occurs in both particulate (0.2-um mean diameter associated with soil particles) and dissolved (or perhaps colloidal) forms. Wet screening and radiometric characterization of the soils showed that a majority of plutonium (and americium) was associated with soils of the 2.4 mm and less size fraction. Attrition scrubbing of soils with hot (80°C) distilled water or aqueous solutions of: chemical agents (e.g., H_2O_2, Na_2CO_3, NaClO, Na_2SiO_3), chelators (e.g., oxalic acid, citric acid), detergents (e.g., Oakite NST), and surfactants (e.g., sodium dioctyl sulfosuccinate) showed varying decontaminating effectivenesses. Oxalic acid (0.1M), sodium hexametaphosphate (10%), and 10% detergent solutions were among the more effective decontaminating systems for the 2.4-4.0 mm soil fraction, removing 95-98% of the Pu and Am. However, residual contamination levels still exceeded 60 dpm/g (27 pCi/g) following the scrubbing process.

Hicks and Blakeslee (1981) also reported soil washing tests on soils from five DOE sites including RFP. Three aqueous solutions: (1) an aqueous pH 12.5, (2) 2% HNO_3, 0.2% HF, 2% pine oil, and 5% hexametaphosphate, (3) 2N HCl were evaluated for decontaminating RFP, Hanford, Mound, INEL, and LANL soils. Variability in the effectiveness of the three-phase scrubbing process was noted for soils from the different DOE sites. Effectiveness of the solutions also varied with the soil size fraction tested.

Pettis and Kallas (1988) conducted bench-scale testing and reported that simple, room-temperature wet screening of 903 Pad soils was effective at decontaminating the greater than 4 mm size fraction (approximately 60 wt% of the total) to <5 dpm/g (2.3 pCi/g) Pu and Am. The >2.4 mm size fraction (approximately 65 wt% of the total) was decontaminated to less than 12 dpm/g (5.5 pCi/g) Pu and 6 dpm/g (2.7 pCi/g) Am by wet attrition scrubbing. The remaining, <2.4 mm soil fraction was treated by attrition scrubbing, ultrasonic scrubbing, oxidation, calcination, desliming, flotation, and heavy-liquid density separation. Although somewhat guarded because of results for selected size fractions, they concluded that attrition or

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vibratory scrubbing, and either mineral jig or acid leaching of this fraction would be effective for a decontamination goal of <30 dpm/g (14 pCi/g).

Steam Stripping

Of the various remediation approaches noted above, under the thermal category of remediation technologies, one innovative technology is dynamic underground stripping— which is an adaptation of steam injection and electrical heating. Steam injection accelerates removal of the NAPL contaminants and is combined with vacuum extraction to perform accelerated removal of volatile contaminants such as underground hydrocarbon spills and electrical heating accelerates the process. Steam injection technology has been demonstrated for remediating NAPLs and VOCs in subsurface soils and clay layers. Aines and Newmark (1992), and Buettner et al. (1992) have successfully tested this technology, in combination with electrical heating, on a bench scale and small field scale for the removal of NAPLs/VOCs in soils or clay layers. EPA's Vendor Information Service for Innovative Treatment Technologies (VISITT) reports that steam stripping technology is being commercialized by Praxis Environmental Services (San Francisco, CA) (Stewart, 1992).

Redox Chemistry

Mobilization of contaminants can obtain as a result of physical or chemical action. Chemically induced mobilization occurs either by chemical action on the contaminant directly (e.g., plutonium) or its support substrate (i.e., soil). In the case of plutonium-contaminanted soils, the mobilization of soil-bound species depends to a large extent on the physical and chemical properties of both the plutonium and the soil.

Plutonium generally exists in three oxidation states: III, IV, and VI, and in the natural environment, plutonium is normally found in either the IV state or, to a lesser extent, the VI state. The oxidation state of the plutonium is a determining factor in its solubility — III and VI states being more soluble than the IV state, whereas,

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americium normally occurs only in the III state. Redox (or chemical *reduction/oxidation*) behavior of plutonium features highly in its propensity for dissolution, and modification of its oxidation state is an important tool in changing solubility. Cleveland (1971) has described conditions for both reduction and oxidation of Pu(IV) using a variety of chemical reagents. We will evaluate simple reducing and oxidizing agents and conditions to accelerate dissolution/mobilization of the plutonium.

Chelation Chemistry

Mobilization of metallic contaminants is greatly enhanced by formation of strong attachments with chemical binding agents via chelation. Chelation chemistry and chelators (or chelates) are often used in association with metals to accelerate dissolution, and/or stabilize/maintain solubility by diminishing the tendency to readsorb or precipitate. Once resuspended or dissolved, the soluble species (in this case radionuclide-chelator complexes) are stabilized by their association with the chelate, which in turn enhances their continued mobility. A variety of complexing or chelating agents, including EDTA, are commonly used for this purpose. Chelating agents can also enhance dissolution by tightly binding the radionuclide and preventing readsorption or precipitation.

Chemically Enhanced Steam Stripping

We propose to marry the technologies of steam injection, redox, and chelation for the leaching/washing of radionuclides from soils. Steam injection/extraction technology, enhanced by redox and chelation chemistry, will be used to mobilize and flush away radionuclides contaminating RFP soils. Conceptually, the process is very similar in design to steam injection systems involving injection and downgradient extraction wells, but with novel modifications to the injection system to provide chemical addition upstream of the soil washing regime.

The technology will be refined to minimize chemical injection with the goal of conducting *in situ* evaluation or *ex situ* treatment with the

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eventual return of the treated soils to the site. The benefits of this approach include: (1) reducing the volume of contaminated soils, (2) avoiding extremely harsh conditions therefore improving the potential for soil post-treatment soil viability and/or replacement, (3) possible tayloring to contaminant and soil conditions, and (4) combining with stream stripping of VOCs. The approach is also potentially applicable to *in situ* operations.

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3.3 Experimental Design and Procedures

3.3.1 Batch Desorption Experiment

Batch desorption experiments involved taking soil samples, adding a solution containing a complexing agent, mixing the two phases, separating the phases, and determining the amount of plutonium and americium in each phase. These desorption experiments were conducted at both 20 °C and 80 °C. All desorption experiments were performed in duplicate.

3.3.2 Column Experiment

Four leaching solutions (sodium citrate, sodium citrate with ascorbate, sodium citrate with dithionite, and sodium chloride solutions) were eluted at 20 °C through a column containing RFP soils. Air dried soil samples were sieved through 2 mm sieve. The soil column experiments was performed in duplicate to ensure the data is reproducible. The amount of radionuclide leached from the RFP soil was determined by total alpha counting. The columns that used contain 350 g of RFP soil. At least ten column volumes were eluted to determine the amount of radionuclides leached from the soil.

3.4 Detail Experimental Procedures

3.4.1 Batch Desorption Experiment

The desorption experiment consists of: 1) weighing 2.5 g of the RFP sieved soil (with a particle size of <53 mm) into an Oak Ridge centrifuge tube, 2) adding 20 ml of a freshly prepared extractant solution to the soil in the tube and capping the tube tightly, 3) placing the tube in an orbital shaker for 24 hours, 4) centrifuging the sample for one hour at 12,000 rpm (28,000 g), 5) decanting the liquid from the solid into a fresh centrifuge tube, 6) centrifuging the decanted liquid for one hour at 12,000 rpm, 7) pipetting 10 mL of the centrifuged liquid into a third centrifuge tube for a final two hour centrifugation cycle at 12,000 rpm, 8) analyzing 5 mL of the centrifuged liquid for ^{239}Pu and ^{241}Am content (using alpha and gamma spectrometry, respectively), 9) weighing the wet solid sample (left from step 5), 10) drying the wet solid under a heat lamp for 48 hours, then weighing again to obtain the weight of the residual extractant liquid from the decanting procedure, and 11) analyzing the dried soil samples for ^{239}Pu and ^{241}Am content using gamma spectrometry.

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Gamma spectrometry analysis was performed using a 180-cc HPGe detector with a well (1 cm in diameter and 5 cm deep) into which a Plexiglas capsule with the sample is placed. This geometry permits capturing about 90% of ^{241}Am gammas and about 60% of the ^{239}Pu gammas. All dry solid samples had a very intense ^{241}Am 59.5 KeV peak and a much weaker, but unmistakably identifiable ^{239}Pu 129.5 KeV peak. The reason why the Pu peak is weaker than the Am one is that only 0.006 alpha-decays of ^{239}Pu are accompanied by a 129.5 KeV photon whereas about half of ^{241}Am decays give a 59.5 KeV photon. Each dry solid sample was measured for about 4 hours. The ^{239}Pu concentration in the solid samples was measured by gamma spectrometry with 20-30% accuracy in most cases; the ^{241}Am concentration was measured with much better accuracy in both solid and liquid samples.

3.4.2 Column Experiment

Column experiments were conducted using the selected desorption solutions. Kontes Chromaflex™ Chromatography glass columns with safety water jackets were used. There are two layers for the column. The inner column was made from glass, and the contaminated Rocky Flats soil was packed into it. The outer column was made from acrylic plastic and used for temperature control. A peristaltic pump was used to pump leaching solution into the soil column.

The size of inner column is 4.8 cm in diameter and 15 cm in length. About 350 g of soil was loaded into the column. about 20 g washed sand was placed on the top and bottom of the column to improve the drainage of washing solution from the soil column. The sand has been washed twice with 5% HCl solution, and once with 30% H_2O_2 solution, then rinsed 3 times with distilled water, and air dried. The flow rate was about 1.5 ml/min. After leaching the column four days with desired solution, the column was washed with 0.1 M sodium chloride. The soil in the column was then removed and air dried. The dried sample was counted for radioactivity by alpha counting. Soil samples were counted at least four times and 20 minutes was used for each counting period. An average of the counting results were reported.

3.5 Sampling and Data Management

Representativeness in the amount of plutonium in the soil to be analyzed is a difficult goal to obtain. It is extremely time consuming to dissolve large amounts of soil prior to alpha spectrometry in order to determine the exact amount of plutonium in each phase. We propose to air-dry and sieve the RFP soils prior to utilization in these experiments. Assuming that the plutonium is associated with a particular size fraction in the RFP soils, choosing a narrow size fraction (that contains a large amount of plutonium) will alleviate the problem of representativeness in sampling and analysis.

All soils received from RFP were stored and handled according to guidelines of "Storage and Handling of Solid Samples."

The data delineated in the procedures were stored in spreadsheets using Microsoft Excel. Hard copies of the spreadsheets which include the data obtained and any procedural deviations will be signed by the technician performing the work and the principal investigator. Electronic copies of the spreadsheets will be stored on a hard disk drive which is backed up on to an optical disk on a weekly basis. All the procedures follow the quality assurance guidelines.

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4.0 RESULTS AND DISCUSSION

4.1 Data Analysis and Interpretation

4.1.1 Batch Desorption Experiment

4.1.2 Column Experiment

Results of column study is shown in the following table.

Leaching Solution	% Removal	Starting pH	Final pH
0.1 M Sodium Chloride	-2%	6.8	6.4
0.1 M Sodium Citrate	20%	6.8	8.3
0.1 M Sodium Citrate with 0.1 M Sodium Dithionite	80%	6.8	6.7
0.1 M Sodium Citrate with 0.1 M Ascorbic Acid	70%	6.8	6.4

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Results suggest that little or no radioactivity was removed from the soil by leaching with 0.1 M sodium chloride solution. However, about 20% of the radionuclides could be washed out by using 0.1 M sodium citrate. Furthermore, about 70% of the radionuclides could be removed from the soil column by using sodium citrate in conjunction with a reducing agent, such as ascorbic acid or dithionite. It should be noted that these radioactivity removal rates are measured by using total alpha counting method and there may be about 15% potential experimental error for using this method.

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Results from column experiments indicate that it is possible to leach at least 70% of radionuclides from contaminated soils by using sodium citrate with reducing agent at room temperature. It is expected that the extracting efficiency of the leaching schemes presented would increase at higher temperatures (such as the ones utilized during steam stripping). Consequently, future experiments should test this hypothesis by conducting leaching experiments at high temperatures using column experiments.

5.2 Recommendations

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6.0 QUALITY ASSURANCE AND CONTROL

Work performed in this project is governed by the Work Plan for Chemical Enhanced Stream Srtipping of Radionuclides in RFP Soils, Quality Assurance Requirements, and applicable Standard Operating Procedures which have been approved by the Los Alamos National Laboratory Quality Assurance Organization.

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